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AN EVALUATION OF IMBEDDED THERMOCOUPLES AS A SOLID-PROPELLANT COMBUSTION DIAGNOSTIC

MARTIN S. MILLER

MARCH 1990

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13. ABSTRACT (Maximum 200 words)

There is a continuing need for temperature profiles through the combustion wave of solid propellants under steady-state conditions. This work aims at assessing the circumstances under which imbedded fine-gauge thermocouples can provide accurate results. Errors can arise from inadequate rates of thermal accommodation to the subject temperature field, from perturbation of the field itself by mismatches in specific heat between thermocouple and propellant or by thermal conduction in the electrical leads from the junction, from catalysis of local reactions at the surface of the thermocouple, and from incomplete thermal accommodation to high gas temperatures because of energy loss by thermal radiation. A systematic approach is adopted wherein each of these sources of error is explored by methodical variation of controlling factors. The study is not yet complete, but preliminary results are presented for two gun propellants at three pressures using 50 micron Pt/Pt10%Rh thermocouples in three lead-angle configurations.

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I. INTRODUCTION

Recently, there has been a renewal of interest in the relatively old technique of mapping reaction-zone temperature profiles in burning solid propellants using imbedded fine-wire thermocouples.[1,2] Combustion-zone temperatures are important both to the long-term goal of modeling the specific chemical reactions involved in propellant combustion as well as to the near-term effort[3] to describe the rate of energy release in a global sense. The imbedded-thermocouple method possesses a number of attractive advantages. Thermocouples can produce temperature profiles through all of the condensed/gas-phase reaction zones in a single trace, and at present are the only means of determining temperature profiles in the condensed-phase portion of the combustion wave. Also, since wires of diameters down to 1 micron have been used, no other existing technique can match their spatial resolution. On the other hand, there are many potential sources of error in such measurements. Response lags, temperature-field perturbation, catalytic heating, and radiation losses are the principal difficulties, but there are a host of other factors, such as failure to achieve steady-state combustion, entrainment of cool shroud gas, inhomogeneities in the propellant sample, etc., which can spoil the data. Although about a dozen groups have reported using imbedded thermocouples over the last 40 years, these technical issues are still not comprehensively resolved.[1] This report describes the approach and fledgling results of a new effort at the Ballistic Research Laboratory aimed at a definitive assessment (and, hopefully, exploitation) of the technique.

The use of imbedded thermocouples in solid propellants was first described by Klein, et al.,[4] who also discussed the errors that can occur in using platinum wires in propellant flames. In addition they recognized the value of the temperature profile in providing the heat-release distribution through the flame. Hunt, Heller, and Gordon[5] showed that, for a double-base propellant at 1 atm, the temperature profile using beaded junctions formed from Pt and Pt-10%Rh wires was unchanged if the wire diameter was decreased from 25 microns to 12.5 microns. This result suggested that the response of 25-micron wire was adequate under the given conditions. Zenin,[6] using ribbon thermocouples (3.5 microns thick x 50 microns wide) of tungsten/rhenium alloys, has produced the most comprehensive body of work to date. Zenin employed the novel (and still unique) idea of imbedding an additional wire probe close to the thermocouple junction and measuring the conduction current between it and one thermocouple leg. Since the electrical conductivity of the polymer increases with temperature and the conductivity of the gas phase is low, the surface temperature on the thermocouple trace was identified by the time at which peak current was measured through the probe. The smallest thermocouples yet used were reported by Kubota, et al.,[7] and consisted of beaded (i.e., flame-welded) junctions constructed from 2.5-micron wire diameter with beads no larger than 4 microns. Since that work, Kubota has successfully employed wires as small as 1 micron in diameter.[8] Suh and Tsai[9] devised a new strategy for determining surface temperature in deflagrating propellants. Recognizing that it may not be possible to use wires small enough to prevent response errors for some propellant formulations and pressures of interest, they attempted to model the condensed-phase response with wire size as a parameter. The model could be used to predict a perturbed temperature profile for various combinations of wire and bead sizes, given an assumed true temperature profile in the propellant. The assumed profile was

then varied until the perturbed-profile predictions matched those actually measured by thermocouples with the same dimensions used in the model. The most successful assumed profile was thereby validated. Most recently, Parr and Parr[10] modeled the condensed-phase response of a thin-ribbon thermocouple (of different material and imbedding configuration than Zenin) and calibrated their heat-transfer coefficient by observing the decay of temperature resulting from a current pulse through the thermocouple under non-burning conditions. They also examined the effect on the response of changing the angle between the thermocouple leads with respect to the direction of maximum gradient in the energetic material during combustion.

The approach envisioned for the present effort is conditioned by an appreciation of the contributions sketched above. First of all, production of a systematic body of data with varying sensor size and angles between the leads is essential as it would immediately enable one to establish the adequacy of the response in the event that the profiles should become invariant below some critical size. Since, predictably, one will want the temperature profile at conditions for which this condition breaks down, this body of data will provide the validation or calibration needed to develop a response model which could be used to extrapolate the finite-sensor results to zero-sized sensors (and, presumably, zero perturbation). Thin but inert coatings must be found to eliminate the catalytic effects expected above about 1000°C[4] without unduly exacerbating the response problem. Zenin's conductivity-probe method appears to be the best means of locating the position of the surface on the temperature trace. An effort should be made to calibrate the thermocouple temperature (corrected for radiation losses where necessary) in regions accessible to other thermometry techniques, e.g., by using spontaneous Raman or line reversal in low-gradient combustion zones of propellants or burner flames. Finally, a technique for thermocouple fabrication should be chosen or developed to provide minimal junction sizes and highly reproducible imbedding configurations in terms of the angle between the leads and orientation to the axis of the propellant strand. The last requirement anticipates the necessity of a response-modeling effort. This report documents the progress to date toward achieving the above objectives.

II. EXPERIMENTAL DETAILS

With regard to the choice of thermocouple for this study, we rejected the use of ribbon types because their advantage is only realized if the combustion front of the propellant is planar and parallel to the plane of the ribbon, these conditions needing to be satisfied to accuracies of the order of a micron for the thinnest ribbons. Several years experience in observing propellant-strand combustion led us to believe that such expectations would be unrealistic. Furthermore, on entering the gas phase, the broadness of the ribbon would likely perturb the gas flow, which is accelerating away from the surface, to a much greater degree than would a wire thermocouple. On the other hand, most wire thermocouples that had been fabricated for this application were flame welded, causing beaded junctions with diameters 2-4 times the diameter of the wire. These large dimensions seemed unnecessarily perturbative. A method for electrically butt welding Type S thermocouples (Pt/Pt10%Rh) by passing DC current through the leads was developed at the Polytechnical University of Milan.[11] A pulsed method (capacitor discharge) had been previously described by Stover.[12] After experience with both techniques we found that a hybrid DC/pulsed procedure worked best. Type S

thermocouples with straight leads and reproducible angles between the leads could be fabricated with butted junctions no larger than the wire diameter. To date, only wires of 50-micron diameter have been made by this method; however, no reason is foreseen to prevent its use with smaller sizes.

Since most finished gun propellant is in the form of small perforated grains, a procedure was developed to resolvate and die-cast the material into the form of a solid cylinder 6 mm in diameter by about 18 mm long. No attempt has yet been made to analyze for residual volatile content above the level of the original grains, but the burning rates of the cast strands and grains are very close as shown in Figure 1a for M30, a triple-base nitrate-ester propellant. The grain burning rates were obtained by inhibiting the perforations with various coatings in an effort to assess their effectiveness in preventing in-perf burning. The least-effective coatings result in the highest pressure exponents because of progressive coning at each perforation. Epoxy was best and produced burning rates very close to those of the resolvated strands. Burning rates for resolvated strands of M30 and XM39 are compared in Figure 1b.

The cast strands were prepared for imbedding by cutting them into pieces as shown in the exploded view of Figure 2a. Using a precision diamond-bladed wafering saw and specially designed chucks, these cuts could be made with great accuracy. Three lead angles (30, 90, 150 degrees), defined as shown in Figure 2b, were considered. Reassembly of the propellant pieces was accomplished in stages: first, one hemi-cylinder was fused to the top cylinder with acetone and dried; next, the resulting piece was placed with the cylinder axis horizontal (imbedding plane horizontal) and manipulated by mechanical stages until properly positioned under the thermocouple; then, the thermocouple, itself held by mechanical manipulators was lowered onto the propellant surface and tacked down with drops of acetone from a 10 microliter syringe; finally, the remaining hemi-cylinder was cemented in place using a propellant/solvent slurry to insure the elimination of voids surrounding the thermocouple. Imbedded samples were cured first at room temperature for 5-6 days then at about 60°C for 6 days.

The samples were burned in a strand burner under constant pressure conditions. An axial shroud of flowing nitrogen prevented the flame from spreading down the sides, making chemical inhibition unnecessary. The burning rate for each run was determined by coordinate-digitizing a time-coded video recording and subjecting the resulting coordinates to least-squares analysis. Figure 3 shows a typical set of coordinate/time data along with the least-squares fit. The degree to which the data fall on a straight line is a measure of the attainment of steady-state combustion, an essential requirement if the temperature profile is to have a well-defined interpretation. The chamber pressure typically varied by a few percent or less during the run, also as seen in Figure 3.

The thermocouple signal, corrected by an electronic ice-point compensator, was boosted by a differential amplifier with a gain of 200 and captured by a digital storage oscilloscope. M30 profiles typically consisted of about 1000 points and XM39 about 5000 points. This data was then transferred via a GPIB to a microcomputer for conversion to absolute temperature, analysis, and display. The signal noise level was typically less than ±2 K.

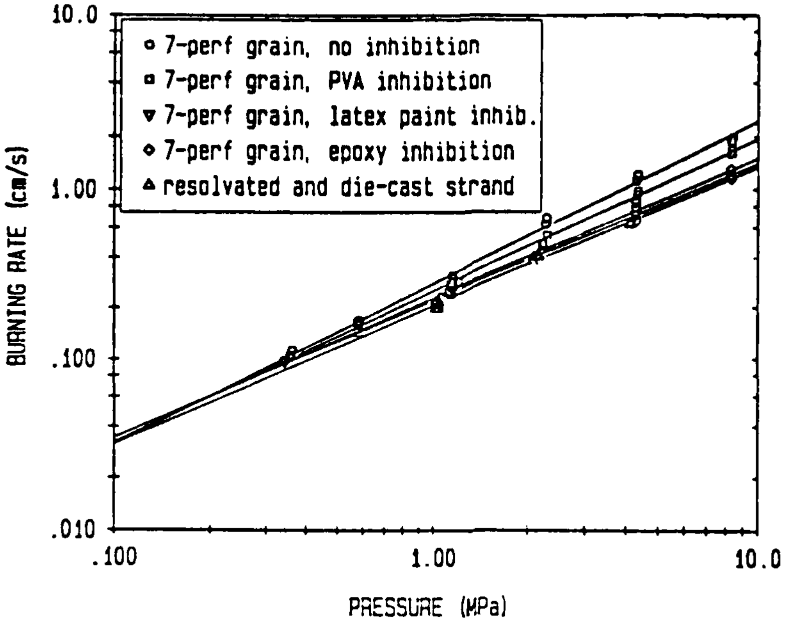


Figure 1a. Comparison of M30 Burning Rates for Resolvated Strands with Those for Perforated Grains Inhibited with Various Coatings

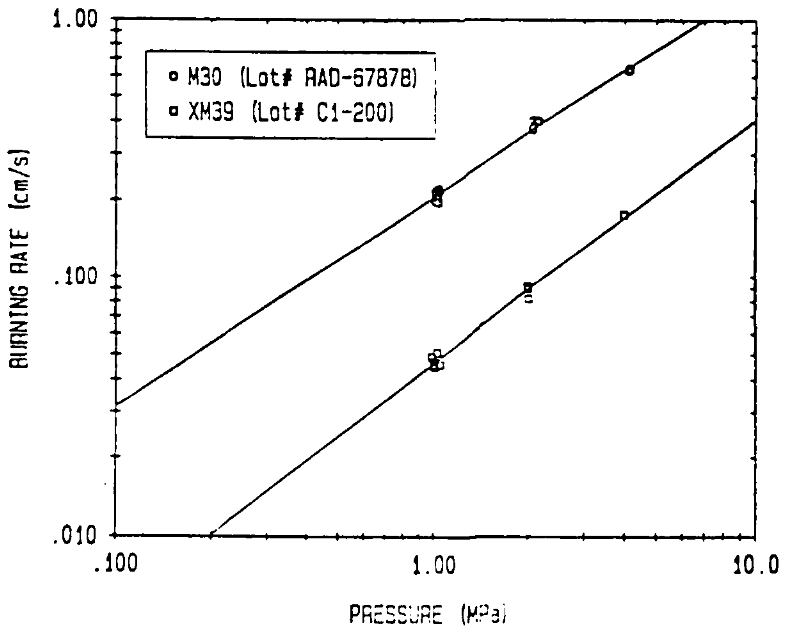


Figure 1b. Burning Rates for M30 and XM39 Resolvated Strands

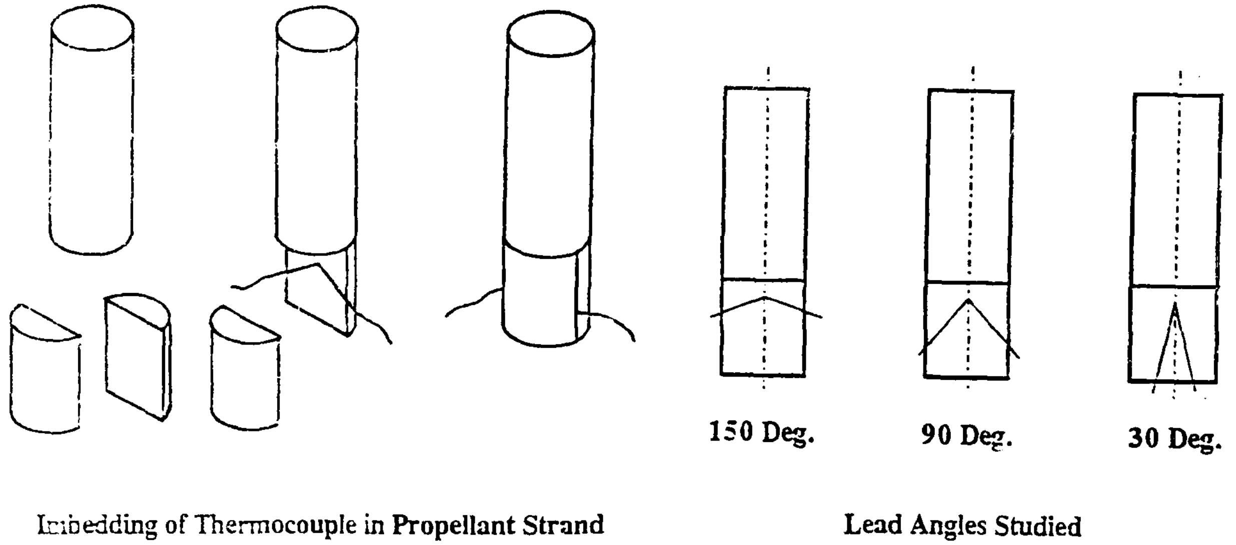


Figure 2. Thermocouple/Propellant-Sample Configuration.
a. Imbedding Steps. b. Lead Angles Defined.

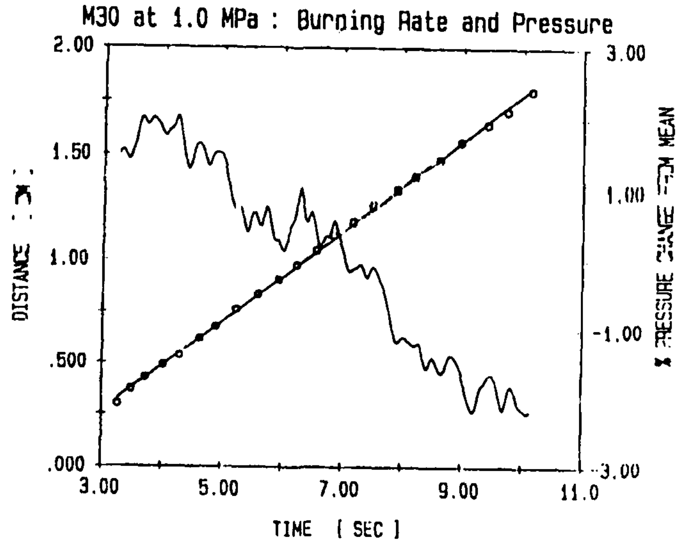


Figure 3. Surface Coordinates as a Function of Time (Points), Least Squares Fit for Burning Rate (Straight Line), and Variation of Chamber Pressure During Run (Wavy Line) for M30 at 1 MPa

III. RESULTS

No attempts have yet been made to determine the location of the surface on the temperature profiles obtained. Although both Zenin[6] and Suh and Tsai[9] have observed a small plateau in the trace at the surface, our larger thermocouples exhibit none. The surface is a natural reference point for comparing one profile to another. Without it there exists a degree of ambiguity in making these comparisons. Worse, computed profile averages can be sensitive to the choice of matching temperature. Here we adopt the convention of matching two or more profiles at a temperature low enough that their responses are, ideally, the same. In practice, if the matching temperature is too low, noise levels or slight differences in emf/temperature relationships can lead to poor matches at higher temperature where these effects are small. This matching problem is exacerbated by flaws in the imbedding process which could result in non-uniformities in thermal diffusivity of the propellant between the junction and the advancing combustion wave. The latter problem, of course, leads to a flawed profile in the condensed phase, and that profile should be rejected altogether. Perhaps the only way to identify these anomalous profiles is by comparison with repeated runs under the same nominal conditions. At this early stage in the present study, however, it has not been possible to identify such anomalies positively.

Figure 4a shows the worst reproducibility among profiles obtained thus far. As experience is gained, valid cause may be found to reject trace S50.29; however, for the purposes of this report, such data will be retained and included in any profile averages displayed. The best reproducibility is illustrated by Figure 4b. Traces are identified by the thermocouple type, "S", followed by the wire diameter in microns, followed by the thermocouple identifying number after the period.

Figure 5 illustrates the effect of lead angle on the thermocouple response. If the plane of the combustion front in the propellant is normal to the strand axis, then the component of the temperature gradient along the thermocouple leads is largest for the smallest lead angles (as defined in Figure 2b), so that these smaller-angles configurations are expected to perturb the temperature field the most by conducting heat away from the junction site. In this figure the 150- and 90-degree curves are each averaged over three individual runs; the 30-degree curve is from a single run since the 30-degree junctions proved to be more prone to breakage.

Only a single run was obtained at 4 MPa for M30 and XM39. These are shown compared to averaged traces at 1 and 2 MPa in Figure 6. Although the true temperature gradient is not resolved by these 50-micron thermocouples, the expected trend of increasing gradient with increasing pressure (burning rate) is evident. For M30 (Figure 6a) the maximum temperature recorded was slightly over 2000 K and was approximately independent of pressure. This compares with an adiabatic equilibrium value (from the BLAKE code[13]) of 2423 K at 1 MPa and 2432 K at 4 MPa. Estimates of the radiation correction using two different heat transfer correlations were less than 50 K. It is not known at present what the cause of this deficit is. One possibility is incomplete reactions, but the relative independence of the measured flame temperature with pressure leads us to discount this explanation in favor of probable cooling by partial mixing with the cool shroud gas. In the future this question will be probed further.

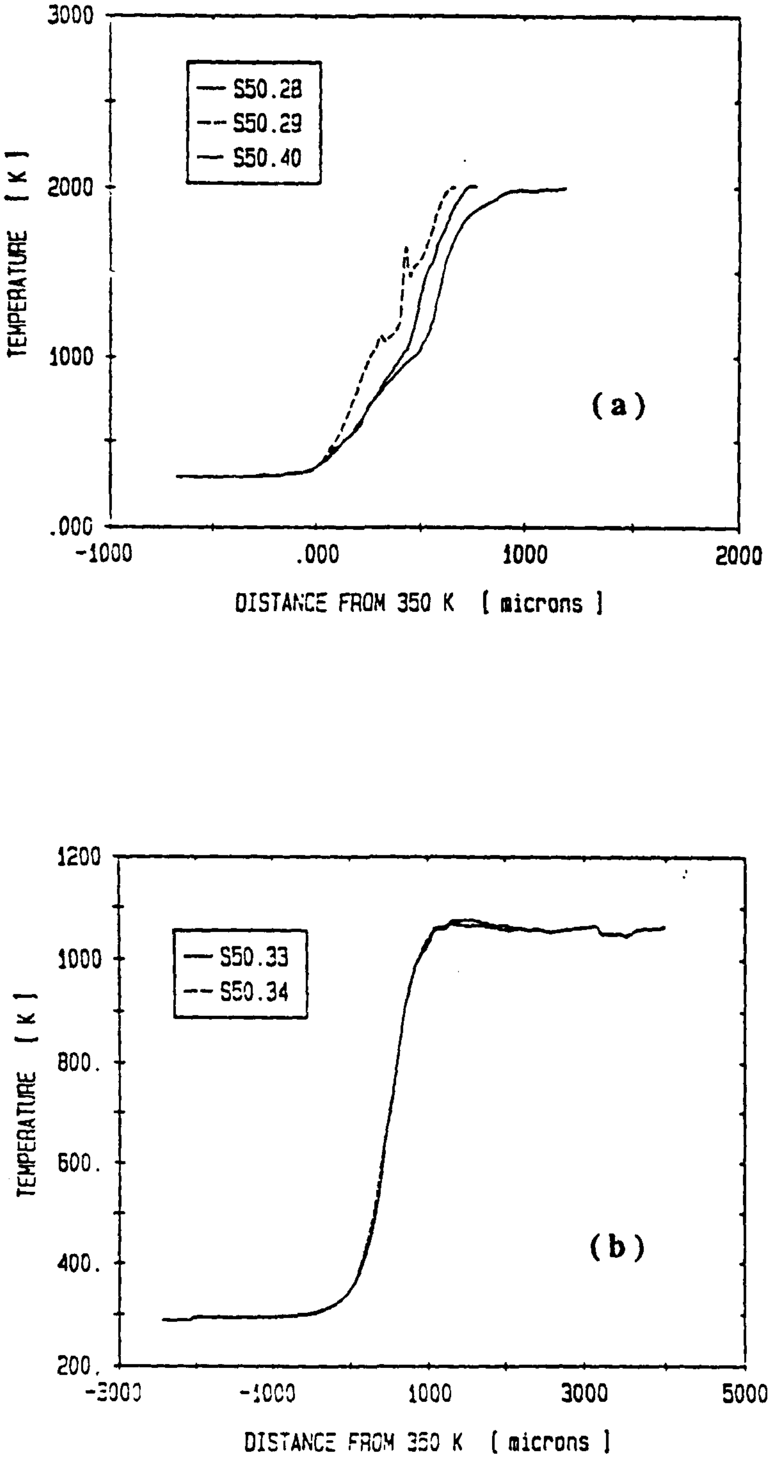


Figure 4. Reproducibility of Temperature Profiles.
a. Worst Case (M30 at 2 MPa). b. Best Case (XM39 at 1 MPa).

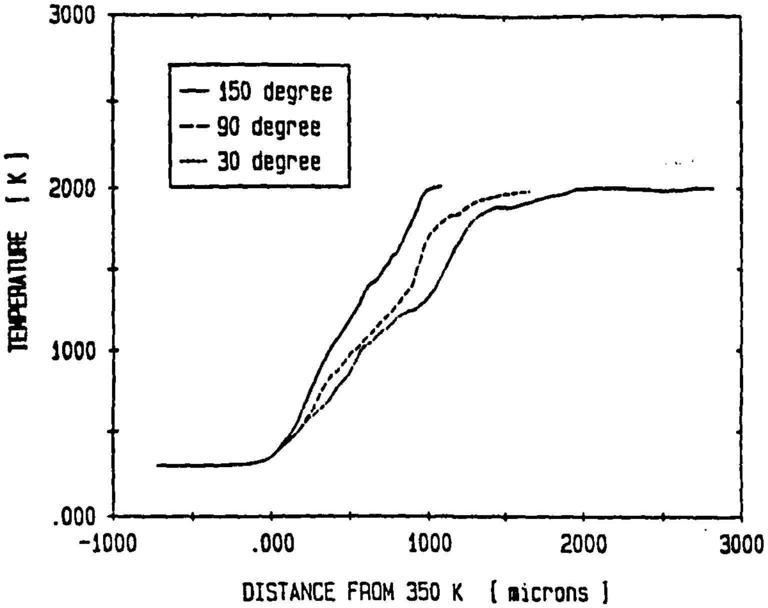
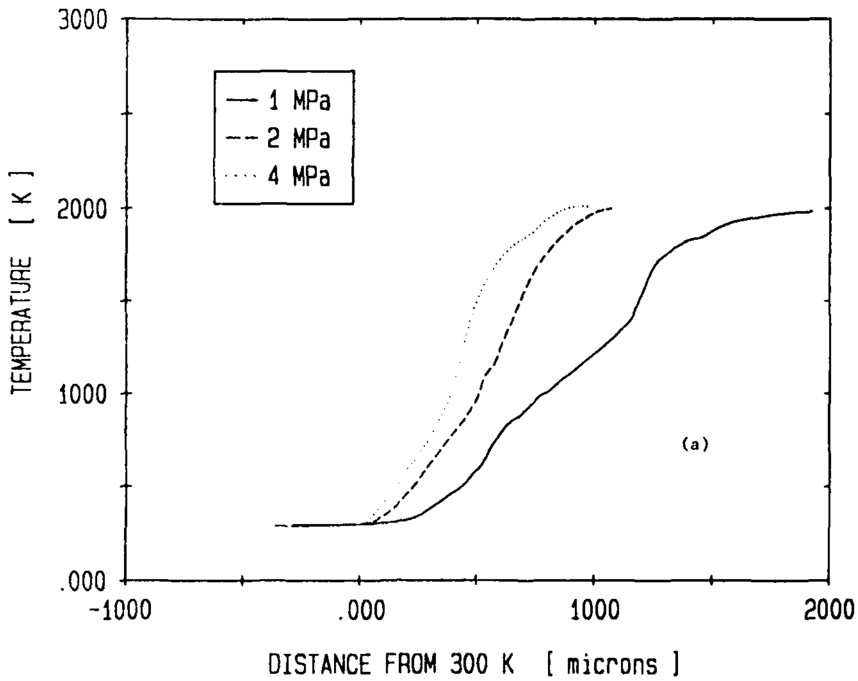


Figure 5. Sensitivity of the Temperature Profile to Variations in Lead Angle (M30 @ 1 MPa)

XM39, on the other hand, exhibits a strong dependence of the measured maximum temperature on pressure. At 1 MPa this temperature was just under 1200 K. Above about 1200 K the averaged 2-MPa profile in Figure 6b gives misleading maximum temperatures. The fluctuating values in the high temperature region is a result of the incipient nature of the visible flame at this pressure. The video record shows this visible flame to alternately attach to and detach from the surface in an unsteady fashion. From an examination of the individual profiles, the maximum temperature measured at 2 MPa was about 1800 K. By comparison the BLAKE equilibrium values[13] are 2214 K at 1 MPa and 2217 at 2 MPa. As the secondary flame is not evident in the video records at 1 MPa, it is probable that the reactions are incomplete at that pressure. At 2 MPa, where the visible flame is weak and intermittent, the measured maximum temperature may fall short of the equilibrium value either because of incomplete reactions or shroud-gas cooling or both. Unfortunately, the 4 MPa profile data was truncated at about 1400 K due to breakage.

As a final example of the preliminary data obtained to date, Figures 7a and 7b compare the averaged temperature profiles for M30 with XM39 at 1 and 2 MPa. Again with the caveat that actual gradients are likely underestimated by the 50-micron thermocouples but assuming relative measurements have some validity, it is interesting to compare the two propellants under the same conditions. At 2 MPa M30 burns about four times faster than XM39, yet the maximum temperature gradient in the near-field reaction zone is about the same. If the surface temperature is in the range 500 to 1000 K, which is likely, then the conductive heat feedback is about the same for the two



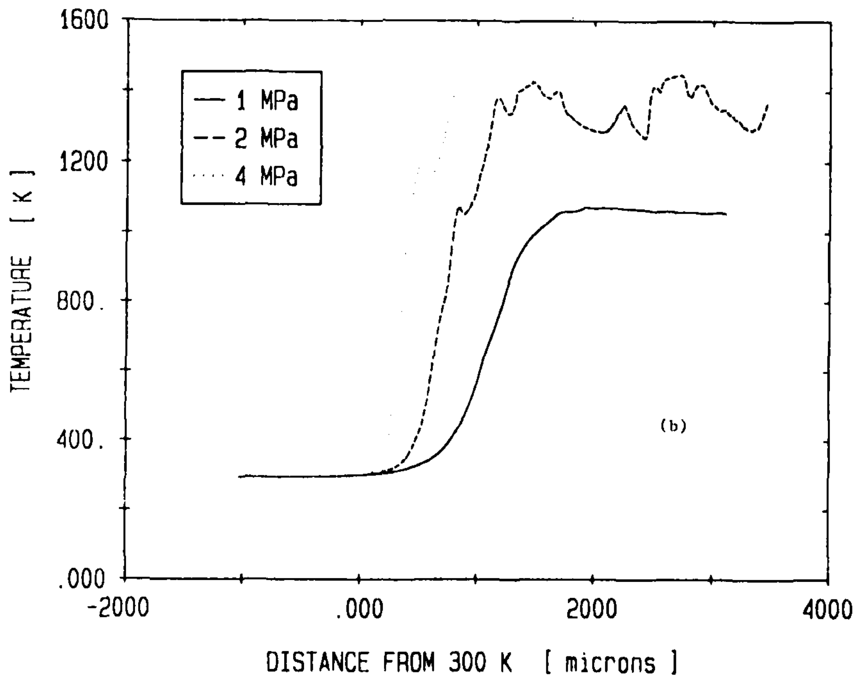


Figure 6. Temperature Profiles for Different Pressures. a. M30 (90° Lead Angle). b. XM39 (150° Lead Angle).

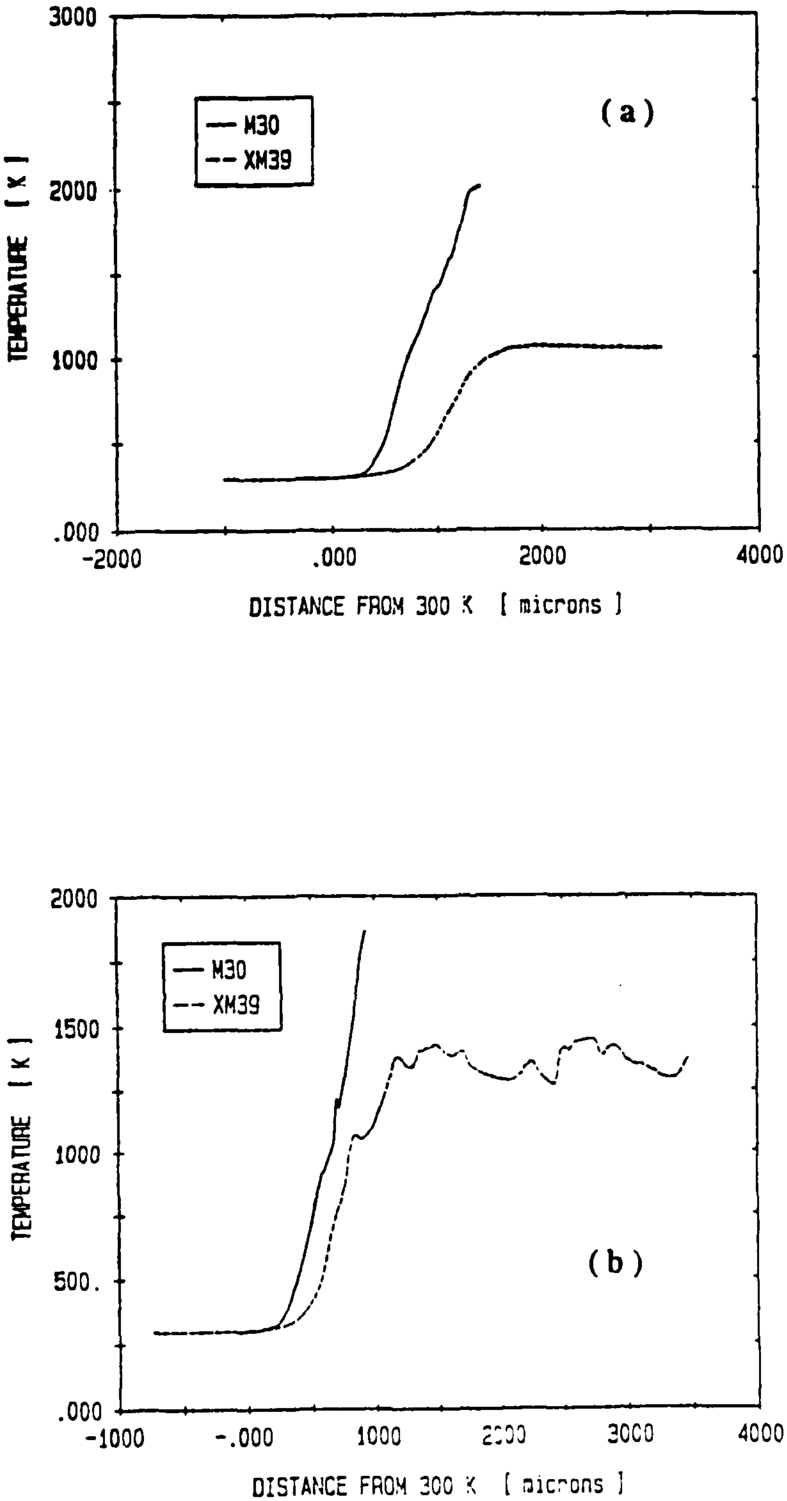


Figure 7. Comparison of Temperature Profiles for M30 and XM39.
a. 1 MPa (150° Lead Angle). b. 2 MPa (105° Lead Angle).

propellants at 2 MPa. If this is the case, then, in the simplest idealization, differences in burning rate must be associated with differences in condensed-phase attributes, i.e., in surface temperature or condensed-phase heat release. A simple expression of the conservation of energy at the surface is[14]

$$\Phi(\mathrm{cal/cm^2\text{-}s}) = Mc_p[T_s - T_o - Q_s/c_p]$$

where Φ is the heat flux fed back to the surface from the gas-phase energy release, M is the mass burning rate, c_p is the specific heat, T_s is the surface temperature, T_o is the initial propellant temperature, and Q_s is the condensed-phase heat release. Assuming the thermal conductivities and specific heats for M30 and XM39 to be approximately the same and equating the temperature gradients at the surface for the two propellants, one obtains

$$4[T_s - T_o - Q_s/c_p]_{M30} = [T_s - T_o - Q_s/c_p]_{XM39}$$

This equation states that the difference between surface temperature and temperature increment due to condensed-phase heat release for M30 must be much smaller than for XM39. The implication is that the surface temperature for XM39 is higher than that for M30, provided that Q_s for XM39 is at least as great as that for M30.

IV. SUMMARY

A new effort has been described with the goal of obtaining accurate temperature profiles through the reaction zones of deflagrating solid propellants using imbedded fine-gauge thermocouples. Previous work has established that such profiles are probably accurate at least under some conditions; however, the general range of validity of the method has yet to be established. The approach developed here seeks to resolve this deficiency through a study involving systematic variation of wire size, lead angle, anti-catalytic coating, propellant type, and combustion pressure. First results are presented for M30 and XM39 at 1, 2, and 4 MPa with three different angles between the thermocouple leads. These measurements were all made with butt-welded Pt/Pt10%Rh wires of 50 micron diameter. Reproducibility of profiles under the same nominal conditions is good in some cases and not in others. Such variability may be due to inconsistencies in the imbedding procedure and will be studied further. Measured secondary-flame temperatures are about 400 K below adiabatic equilibrium flame temperatures for reasons that are as yet unclear; this matter will also be studied further. At 1 MPa XM39 burns without a visible flame with a maximum temperature about half the adiabatic equilibrium value, most likely as a result of incomplete reactions. Continued work will focus on decreasing the size of the thermocouple, locating the surface on the trace using Zenin's method, examining various anti-catalytic coatings, and calibrating the high-temperature measurements using laser Raman spectroscopy.

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